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**MS-2-P-2786 Bilayer graphene structures formed by passage of current through graphite: HRTEM and HAADF-STEM studies**

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The subject of this paper is a new form of carbon which can be formed by passing an electric current through graphite [1,2]. This new carbon apparently consists of hollow graphitic shells bounded by curved and faceted planes, typically made up of two graphene layers. We describe studies of this carbon using high resolution transmission electron microscopy (HRTEM) and high-angle annular dark-field scanning transmission electron microscope imaging (HAADF-STEM). These studies appear to confirm that the bilayer graphene structures are 3-dimensional.

The carbon was prepared in an arc-evaporator which is normally used for coating specimens for scanning electron microscopy. Following evaporation, a small deposit was formed in the area where the two graphite electrodes made contact, and it was this deposit which contained the “transformed” carbon.

Some conventional TEM images of the transformed carbon are shown in Fig. 1. In the low magnification image (Fig. 1(a)), it can be seen that the outline of the structure is much more irregular than in normal graphite, with many curved and unusually-shaped features. Higher magnifications images, such as Fig. 1(b), show that the transformed carbon consists largely of bilayer graphene.

In order to determine the 3-dimensional shapes of the graphene structures we have used HAADF-STEM imaging. Both individual images and tilt sequences have been analysed. Individual HAADF-STEM images were recorded on an aberration-corrected Nion UltraSTEM100, operated at 60kV. Figure 2(a) shows a HAADF-STEM image of a region in which a nanotube is joined to a larger bilayer structure. The contrast in this image, in combination with a quantitative analysis of the near edge fine structure of the C K EELS edge [4], indicate that the edges of the structure are highly curved. This is consistent with the 3-dimensionality of this material.

Tilt series were recorded using an FEI Titan microscope operated at 80kV. A typical tilt sequence is shown in Fig. 2(b). This appears to show a 3-dimensional particle with the shape of a flattened cone.

Structural transformation of graphite as a result of the passage of an electric current has been observed by other groups [e.g. 5,6]. These groups have discussed the process in terms of the sublimation and edge reconstruction of flat graphene. However, as argued here, there are good reasons for believing that the transformed carbon is in fact 3-dimensional. If this is correct, this new carbon may have a number of possible applications, for example in supercapacitors.


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Fig. 1: Conventional HRTEM images showing structure of carbon following passage of current.

Fig. 2: HAADF-STEM images of structures in transformed carbon. (a) Image showing junction between bilayer nanotube and larger region, (b) tilt sequence of approximately conical structure.